Designation: D1603 – 14

Standard Test Method for Carbon Black Content in Olefin Plastics[1]

This standard is issued under the fixed designation D1603; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers the determination of the carbon black content in polyethylene, polypropylene, and polybutylene plastics. Its use with acrylic or other polar monomer modifications which might affect the accuracy is not recommended. Determinations of carbon black content are made gravimetrically after pyrolysis of the sample under nitrogen. This test method is not applicable to compositions that contain nonvolatile pigments or fillers other than carbon black.

1.1.1 This test method is not applicable to materials containing brominated flame retardant additives at the end.

1.2 The values stated in SI units are to be regarded as standard. The values in parentheses are given for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—This standard and ISO 6964-1986(E) address the same subject matter, but differ in technical content.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

D4218 Test Method for Determination of Carbon Black Content in Polyethylene Compounds By the Muffle-Furnace Technique

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Standard:*

ISO 6964-1986(E) Polyolefin Pipes and Fittings—Determination of Carbon Black by Calcination and Pyrolysis—Test Method and Basic Specification[3]

3. Terminology

3.1 *Definitions*—For definitions of technical terms pertaining to plastics used in this specification, see Terminology D883.

4. Significance and Use

4.1 The information provided by this test method is useful for manufacturing quality control, technical service, and research purposes; and is required by various material specifications and for the calculation of optical absorptivity.

4.2 Test Method D4218 is available for determining the carbon black content of polyethylene compounds if so desired.

5. Apparatus

5.1 *Electric Furnace,* at least 20 cm (7.9 in.) long suitable for use with the tubing described in 5.2.

5.2 *High Temperature Glass Combustion Tube*[4], of appropriate diameter and approximately twice as long as the furnace described in 5.1.

5.3 *Stoppers*—Two rubber or neoprene stoppers, to fit the tube described in 5.2, unless the tube is fitted with ground joints and mating connectors.

5.4 *Glass Tubing,* approximately 10 mm (0.39 in.) in diameter, of sufficient amount, and matching rubber or plastic tubing for connections.

5.5 *Combustion Boat,* approximately 8 by 1.9 by 1.3 cm (3.15 by 0.75 by 0.51 in.). Glazed porcelain, quartz high-silica glass, or platinum is suitable.

NOTE 2—A loose-fitting cover for the combustion boat is optional. If

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.01).

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] *ISO/IEC Selected Standards for Testing Plastics, Second Edition*, published by ASTM. Also available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

[4] Borosilicate, high-silica, or equivalent glass tubing has been found satisfactory for this purpose.

***A Summary of Changes section appears at the end of this standard**

used, it shall be considered a part of the boat and handled and weighed with it.

5.6 *Iron-Constantan Thermocouple,* and a potentiometer or millivoltmeter suitable for determining temperatures in the range 300 to 700°C (572 to 1292°F).

5.7 *Flow Meter,* suitable for measuring gas flow at rates of 1 to 10 L/min.

5.8 *Traps,* three glass traps with removable ground-glass connected heads and 10-mm (0.39-in.) diameter inner and connecting tubes.

NOTE 3—Only one trap is required if the entire apparatus train is placed in a fume hood. None is required if in addition, nitrogen of sufficient purity is used and produced by the alternative means provided in Section 6.

5.9 *Drying Tube*—A U-shaped drying tube, having an inside diameter of 20 mm (0.79 in.) or larger, fitted with ground glass or neoprene stoppers.

5.10 *Glass Wool.*

5.11 *Desiccator,* with desiccant.

5.12 *Bunsen Burner or Muffle Furnace*—Electric resistance-heated or microwave-heated furnace capable of heating the combustion boat to red heat.

NOTE 4—When an electric furnace is used, position it in a well-ventilated hood. When a microwave furnace is used, position it within or adjacent to a hood and the exhaust tube vented into the hood to prevent the breathing of byproducts of any combustion. An air flow rate of 2.8 m^3/min through the microwave oven is recommended.

5.13 *Balance*—An analytical balance having a sensitivity of 0.0001 g.

6. Reagents and Materials

6.1 *Carbon Dioxide, Solid* (Dry Ice).

NOTE 5—The solid carbon dioxide and the trichloroethylene are not required if the entire apparatus train is placed in a fume hood.

6.2 *Desiccant,* such as anhydrous calcium chloride ($CaCl_2$).

6.3 *Nitrogen,* prepurified, having oxygen content below 0.01 %. As a safeguard against accidental leakage, contamination, or inadequate purity, the gas shall be further purified by one of the following procedures:

6.3.1 Passage of the nitrogen through a glass trap inserted ahead of the drying tube (see Fig. 1), filled approximately one third full of potassium hydroxide - pyrogallol solution made to contain 5 g of pyrogallol and 50 g of KOH in 100 mL of water. Technical grade, or better, reagents are satisfactory.

6.3.2 Insertion of a plug, or roll, of clean copper tinsel, foil, or wire 7.5 to 10 cm (3 to 4 in.) long into the combustion tube ahead of the sample (see Fig. 1) so that it is completely within the heated region of the furnace. Take care to prevent channeling of the nitrogen through the plug. The extent of blackening of the copper may be taken as a guide for determining when the plug should be renewed.

6.3.3 Passage of the nitrogen through a combustion tube filled to a length of 15 cm (6 in.) or greater with clean copper tinsel, foil, or wire, and maintained in a furnace at a temperature around 500°C (932°F).

6.3.4 The need for the procedures described is eliminated if gas having an oxygen content of less than 0.002 % (20 ppm) is used.

6.4 *Trichloroethylene,* technical grade (Note 5).

7. Sampling and Test Specs

7.1 The test specimens can be in a variety of forms which fit in the combustion boat but must satisfy the requirements of 8.3. Soiled articles must be washed and printed articles are wiped clean with a suitable solvent.

8. Procedure

NOTE 6—The procedure below assumes that the combustion tube can be easily removed from the furnace. If this is not the case, alternate methods of inserting and removing sample boats are acceptable as long as the

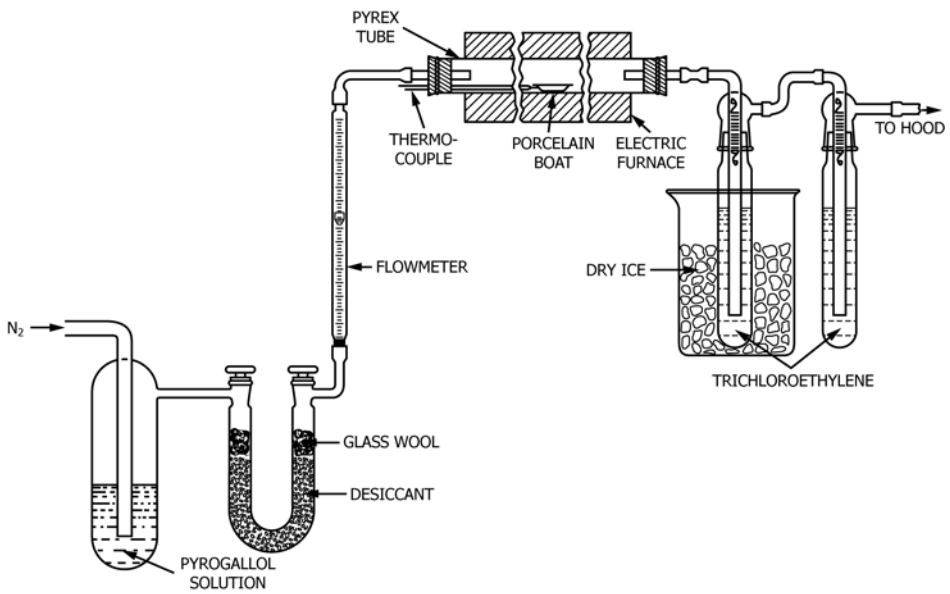

FIG. 1 Assembly of Apparatus

temperature, purge time, and flow rate requirements are met.

8.1 Assemble the apparatus as shown in Fig. 1. Both cold traps following the combustion tube shall contain trichloroethylene, but only the first need be cooled with solid carbon dioxide. Alternatively, the entire apparatus may be placed in a fume hood and the two traps following the combustion tube omitted. Fill the drying tube with anhydrous $CaCl_2$ or other suitable desiccant. Hold between loose plugs of glass wool.

8.2 Heat a clean combustion boat to red heat in a bunsen flame or muffle furnace; then transfer the boat to the desiccator and allow it to cool over fresh desiccant for not less than 30 min.

8.3 Remove the boat from the desiccator and weigh it to nearest 0.0001 g (w_1). Immediately place 1.0 ± 0.1 g of the ethylene plastic under test in the boat and quickly weigh to the nearest 0.0001 g (w_2).

8.4 Heat the furnace to a constant temperature of 600°C (1112°F).

8.5 With the combustion tube removed from the furnace, adjust the rate of nitrogen flow through the tube to 1.7 ± 0.3 L⁄min. Open the inlet end of the combustion tube, quickly place the combustion boat with the sample into the tube positioned so the boat will be at the proper temperature when in the furnace. Close the inlet to the tube and allow the nitrogen to flow for a minimum of 5 min to purge oxygen from the system prior to placing the tube in the furnace. If the furnace is controlled by an external thermocouple, adjust the thermocouple so that the weld is in proximity to the boat. Insert the copper plug, if this is used (see 6.3.2). Quickly place the tube into the furnace, close the furnace, and allow heating to proceed for at least 15 min.

NOTE 7—The exact temperature of heating is not critical in the range 500 to 700°C (932 to 1292°F), although a heating time as long as 30 min is desirable at the lower temperature. If desired, the sample may be put in the furnace at 300°C (572°F) or less and the temperature of the furnace then programmed for sample heating to 350°C (662°F) in 10 min, 450°C (842°F) in another 10 min, and 500°C (932°F) after a total of 30 min, finally heating at 500°C (932°F) for an additional 15 min.

NOTE 8—If the combustion tube and furnace are of adequate size, several sample boats may be tested simultaneously if they are positioned in the furnace within the area which meets the temperature requirements.

8.6 Move the tube or furnace so that the boat is no longer in the heated zone of the furnace and allow 5 min for cooling, while maintaining the flow of nitrogen. Remove the copper plug, if present, and the boat through the inlet end of the tube and allow it to cool in the desiccator for at least 30 min. Take care that the boat does not become contaminated from any deposits on the walls of the tube. Then quickly reweigh the boat and its contents to the nearest 0.0001 g (w_3).

8.7 If carbon black measurements are to be made at values less than 1 %, then the sample boat with the residue of the tube furnace burn shall be placed in a muffle furnace for approximately 10 min to oxidize the carbon black. This is to correct for the presence of residual inorganic matter in the sample. After heating, cool the sample boat in a desiccator until it is at room temperature. Weigh the boat plus contents to the nearest 0.0001 g (w_4).

8.8 Two determinations are made for each sample.

9. Calculation

9.1 Calculate the carbon black content as follows:

$$\text{Carbon black, \%} = \frac{(W_r - W_o)}{W_s} \times 100 \quad (1)$$

where:

W_r = $w_3 - w_1$ = mass of residue (g) after burning in nitrogen,
W_o = $w_4 - w_1$ = mass of residue (g) after burning in air, and
W_s = $w_2 - w_1$ = mass of sample (g).

10. Report

10.1 Report the following information:

10.1.1 Complete identification of material tested, including type, source, manufacturer's code number, form, previous history, etc.

10.1.2 If applicable, the specific location of the specimen, if significant, for example, from articles.

10.1.3 The individual determinations calculated as described in Section 9, and

10.1.4 The average of the determinations reported in 10.1.3.

10.1.5 Date.

11. Precision and Bias[5]

11.1 The precision of this test method is based on an interlaboratory study of this test method conducted in 2011. Five laboratories tested three different olefin plastic materials. Every "test result" represents the average of two determinations. Each laboratory reported three replicate test results for each material. Except for the limited number of participating laboratories, Practice E691 was followed for the design and analysis of the data.

11.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the *r* value for that material; *r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

11.1.1.1 Repeatability limits are listed in Table 1.

11.1.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the *R* value for that material; *R* is the interval representing the critical

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1256.

TABLE 1 Carbon Black Content (%)

Material	Average[A]	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{x}$	s_r	s_R	r	R
Material A	1.74	0.03	0.18	0.09	0.50
Material B	5.27	0.08	0.16	0.21	0.45
Material C	0.79	0.01	0.07	0.03	0.20

[A] The average of the laboratories' calculated averages.

difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

11.1.2.1 Reproducibility limits are listed in Table 1.

11.2 The terms repeatability limit and reproducibility limit are used as specified in Practice E177.

11.3 Any judgment in accordance with statements 11.1.1 and 11.1.2 would normally have an approximate 95 % probability of being correct, however the precision statistics obtained in this ILS must not be treated as exact mathematical quantities which are applicable to all circumstances and uses. The limited number of laboratories reporting replicate results guarantees that there will be times when differences greater than predicted by the ILS results will arise, sometimes with considerably greater or smaller frequency than the 95 % probability limit would imply. Consider the repeatability limit and the reproducibility limit as general guides, and the associated probability of 95 % as only a rough indicator of what can be expected.

11.4 *Bias*—At the time of the study, there was no accepted reference material suitable for determining the bias for this test method, therefore no statement on bias is being made.

11.5 The precision statement was determined through statistical examination of 45 results, from five laboratories, on three different olefin plastic materials described as:

Material A: LLDPE Polyethylene
Material B: LLDPE Polyethylene
Material C: HDPE Polyethylene

12. Keywords

12.1 carbon black; content; gravimetric; plastics; polyolefins

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue, D1603 - 12, that may impact the use of this standard. (August 1, 2014)

(1) Corrected the calculation for % Carbon Black in Section 9, Calculation.